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PREPARATION OF OsB_2 BY ARC-MELTING

Paul G. Curtis

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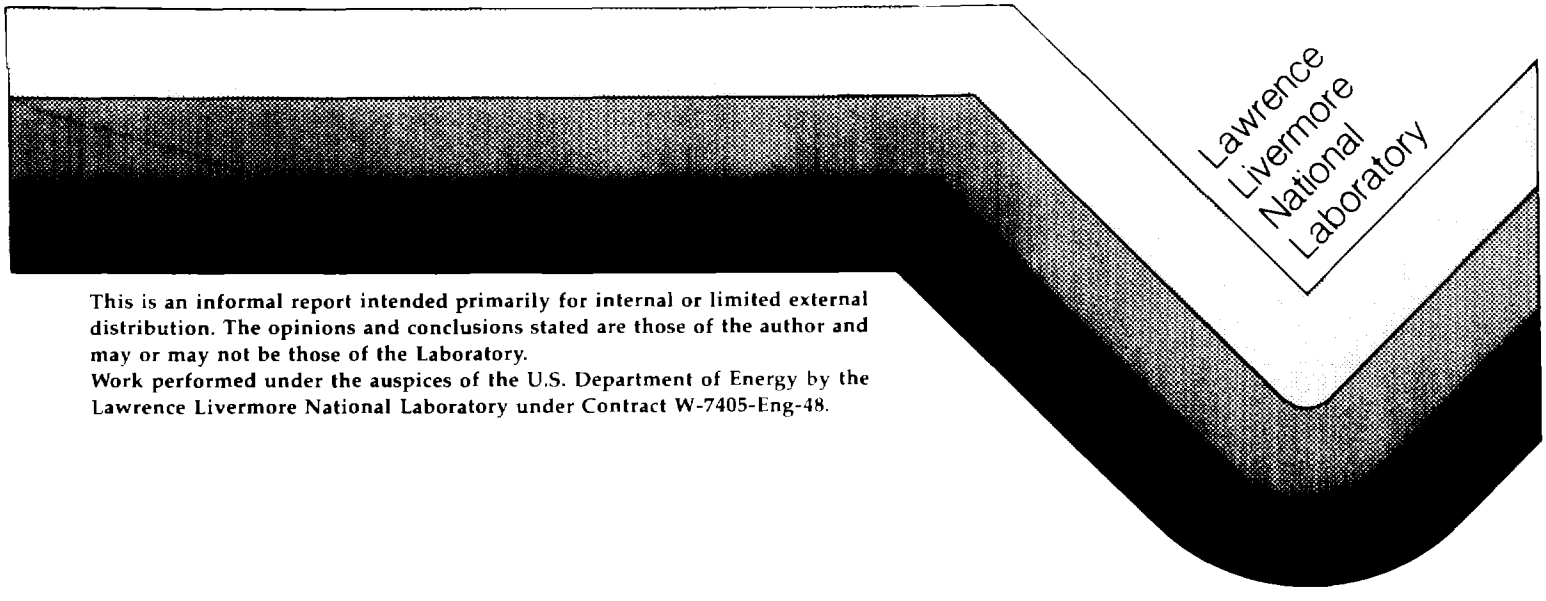

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Purpose

The purpose of this work was to develop an arc-melting method to form osmium diboride (OsB_2) in powder form.

Starting Materials

A high purity Os powder from Custom Chemical Company was supplied by the requestor (Winifred Burks). Crystalline boron of 99.8% purity from United Mineral and Chemical Company was used from stock we had on hand. The boron was in chip form approximately 1-3 mm in size.

Description of Arc-Melter

The arc-melter is built into a glove box containing a water-cooled copper hearth, welding cables and a hand-held electrode holder. A high-current DC power supply for the arc is located outside the glove box. A current range setting of 19-252 amps was used for the Os-B melts.

The heat for melting is produced by setting up an arc between a non-consumable thoriated tungsten electrode (the anode) and the material to be melted (the cathode). The electrode, melt, and arc are protected from contamination by an argon atmosphere in the glove box. Before the Os and B are melted together, a Ti ingot is melted. The Ti is an oxygen getter. If the Ti ingot shows any tarnish upon cooldown, this indicates that the Ar contains too much oxygen to melt without oxygen contamination. Any air or water leaks in the glove box will therefore have to be fixed before continuing with the arc-melting operation.

Exploratory Experiments

Before arc-melting the Os and B together, a test melt was done with a mixture of W powder and B chips to give a WB_2 stoichiometry. During the melt the arc beam tended to deflect away from the B, and the B frequently popped (fractured) and spattered. This problem was solved by heating up the B very slowly until it became conductive (resistivity decreases with temperature). Once the B became conductive, enough power could be applied to the arc to melt the B and W mixture.

For the initial OsB_2 melt, Os powder and B were mixed together in the proper proportion in a hearth and melted. During melting, a large part of the Os powder was blown out of the hearth, most likely because of outgassing of the Os powder. To overcome this problem, the Os starting powder was arc-melted into an ingot. The ingot was then crushed with a diamond platner into chips approximately 1-3 mm in size. The Os chips were then combined with the B as before and arc-melted this time with satisfactory results.

Preparation of OsB_2

Premelted Os metal chips, approximately 1-3 mm in size, were combined in the proper proportion with crystalline boron chips of the same size to obtain a final ingot of 10 grams. The mixture was arc-melted in an argon atmosphere in a water-cooled copper hearth. The OsB_2 ingot was remelted six times to insure a homogeneous mixture of the alloy. For each remelt, the ingot was was allowed to cool and then turned over and melted.

To prepare the OsB_2 powder, the 10 gram ingot was crushed into small chips with a diamond platner. The chips were then pulverized into a fine powder in a

vibratory mill, called a Speck Mill. The Speck Mill was run for two hours to obtain a sufficiently fine powder. The sample container (50 ml capacity) for the mill was lined with tungsten carbide and used two tungsten carbide balls for grinding.

Results and Discussion

X-ray diffraction results of the powder showed only the pattern for the OsB_2 phase. The powder was sieved and found to range in size from <10 to 90 μm. The majority of the powder was between 20-40μm. We found the Os in powder form was very difficult to work with. We solved this by melting the powder into an ingot, and then crushing it into small chips. The chips were then blended with the boron. In the future work it would be helpful if the Os could be supplied in chip or foil form. This would save melting the powder into an ingot and the crushing it prior to combining with B.